Los Alamos National Laboratory **Environmental Restoration Program** Standard Operating Procedure	No: LANL-ER-SOP-09.06 Rev: 0

Zeolite Purification and Separation

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ZEOLITE PURIFICATION AND SEPARATION

1.0 PURPOSE

The purpose of this procedure is to describe the methods, procedures, and documentation used to purify/separate zeolites from zeolite-rich samples by sedimentation.

2.0 SCOPE

2.1 Applicability

This procedure applies to zeolite samples purified/separated for the Environmental Restoration (ER) Program.

2.2 Training

Only those persons certified for this procedure shall perform ER-related zeolite purifications/separations. Reading and understanding this procedure and the procedures in Section 1.0, General Instructions shall constitute training to this procedure.

3.0 DEFINITIONS

N/A

4.0 BACKGROUND AND/OR CAUTIONS

Zeolite-rich samples can be purified or extracted from a sample by means of sedimentation in water. This method relies on the difference in particle sizes and densities of the mineral components in the sample. This method is preferred over other methods due to the low magnetic susceptibilities of the zeolite (magnetic separations) or where one is concerned with potentially altering the chemical composition of the zeolite (heavy-liquid density separations).

Purity of the zeolite fractions may be confirmed by X-ray diffraction analyses. It is up to the certified personnel conducting the separation to decide whether the zeolite has been purified/separated sufficiently for the work to be conducted. This decision shall be documented in the Daily Activity Log, (see SOP-01.01.01, Records), by the certified personnel before conducting further research with the sample.

Known Limitations:

- This technique requires that the initial sample be fairly rich in zeolite.
- Minerals intergrown with the zeolites (such as opal-CT in clinoptilolite) can not be separated.

- Two similar species of zeolites such as mordenite in clinoptilolite do not separate very well.

5.0 EQUIPMENT

- Shatterbox (or large ball mill) -- most all acceptable.

- Ultrasonic probe capable of ~200 W.

  The Ultrasonic probe is placed in a sound-dampening box to help protect the hearing of the operator. The use of de-ionized or distilled water is necessary to ensure that no chemical alteration of the zeolites occurs due to cation exchange. Thoroughly clean all sample preparation equipment and work areas before use.

6.0 PROCEDURES

Note: Calibration is not applicable. Samples are examined for purity using X-ray powder diffraction in accordance with SOP-09.04.

6.1 Samples

6.1.1 Samples will be tracked, handled, shipped, and stored in accordance with the procedure for Sample Identification and Control for Mineralogy-Petrology Studies (Ref A, SOP-01.03, Handling, Packaging and Shipping of Samples, and SOP-01.04, Sample Control and Documentation).

6.1.2 Carefully label containers into which the sample separates will be placed. Label sides and top of each container so that tops cannot be switched.

6.2 Separation Procedure

6.2.1 The zeolite-rich sample is first shatterboxed or ball-milled (~1 to 2 minutes) to break the sample down to a particle size that is closer to the grain size of its constituent phases.

6.2.2 ~30 to 60 grams of the zeolite sample are placed in a 1000ml plastic beaker filled with ~700-800ml of deionized water. (Glassware, beakers, and settling times are to be scaled appropriately when working with amounts of sample smaller than 30-60 grams).

6.2.3 Disaggregate the sample for approximately 10 to 15 minutes at ~200 W using an ultrasonic probe. Longer times may be used if samples are difficult to disaggregate.

6.2.4 Place the sample on a vibrationally stable surface without cooling it (the ultrasonic probe generates heat in the suspension) and allow it to settle for 30 to 60 seconds. The sediment is composed of the coarse fraction (>~20mm) which should include much

of the quartz, feldspars, and other mineral impurities, and also any larger clumps of zeolites that were not disaggregated. This fraction may be discarded.

6.2.5 Decant or syphon the supernatant into a second beaker and allow it to rest on a vibrationally stable surface for ~1 hour to settle out the 20-30mm size fraction. This is generally a fairly pure zeolite fraction.

6.2.6 Decant or syphon the supernatant into a third beaker and allow it to rest on a vibrationally stable surface overnight (15-20 hours) to settle out the ~3.0-1.0mm size fraction. This should also be a rather pure zeolite fraction.

6.2.7 The remaining supernatant, although it may have a high concentration of zeolite remaining, generally has a high concentration of smectite. It can be discarded, or further processed to concentrate the smectite for clay mineral analysis using Clay Mineral Separations, SOP-09.06.

6.2.8 The sediments from 6.2.5 and 6.2.6 are dried by placing the beakers with the sediments on a warm surface such as a hotplate set at ~50°C or allowed to air-dry at room temperature.

6.2.9 The dried sediments may be X-rayed to determine their purity in accordance with Operation of Siemens X-Ray Diffraction, SOP-09.04. Steps 6.2.2 to 6.2.7 may be repeated if further purification/separation is desired.

6.3 Procedural Deviations

Deviations from this procedure shall be fully documented in the Daily Activity Field Log of the person conducting the purification/separation explaining the deviation and the effects it may have on the resulting work.

7.0 REFERENCES

Los Alamos National Laboratory Yucca Mountain Project Quality Assurance Manual in effect 1/89, Sample Identification and Control for Mineralogy- Petrology Studies.
LANL-ER-SOPs in Section 1.0, General Instructions
LANL-ER-SOP-09.03, Operation of Siemens X-Ray Diffraction.
LANL-ER-SOP-09.05, Clay Mineral Separation.

8.0 RECORDS

All zeolite-rich samples purified/separated using this procedure shall be documented on Daily Logs including complete sample name or number, date that each sample was processed, and the signature of the person conducting the separation.

All documentation requirements imposed by other procedures, both Environmental Restoration and Yucca Mountain Project, used during this procedure shall be completed in accordance with the procedure.

Separated/purified zeolite fractions shall be placed in appropriately labeled sample bottles.

The notebook entry for a sample shall constitute evidence that the procedure has been implemented and satisfactorily accomplished for that sample.

9.0 ATTACHMENTS

N/A